

The atomic scale investigation of Nb for superconducting RF cavity

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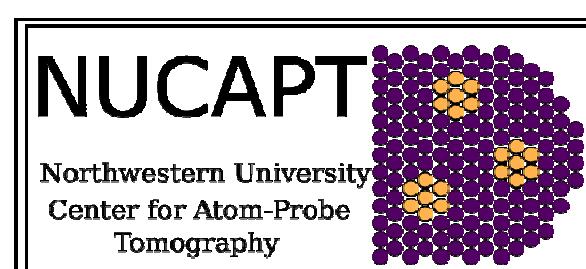
- Northwestern University

Pierre Bauer, Chris Boffo

- Fermi Lab

Jim Norem

- ANL



Outline

- Oxygen in Nb
- Sample preparation
- Atom Probe tomography (APT)
- Result from Local Electrode Atom Probe (LEAP) tomograph
- Conclusions
- Next step

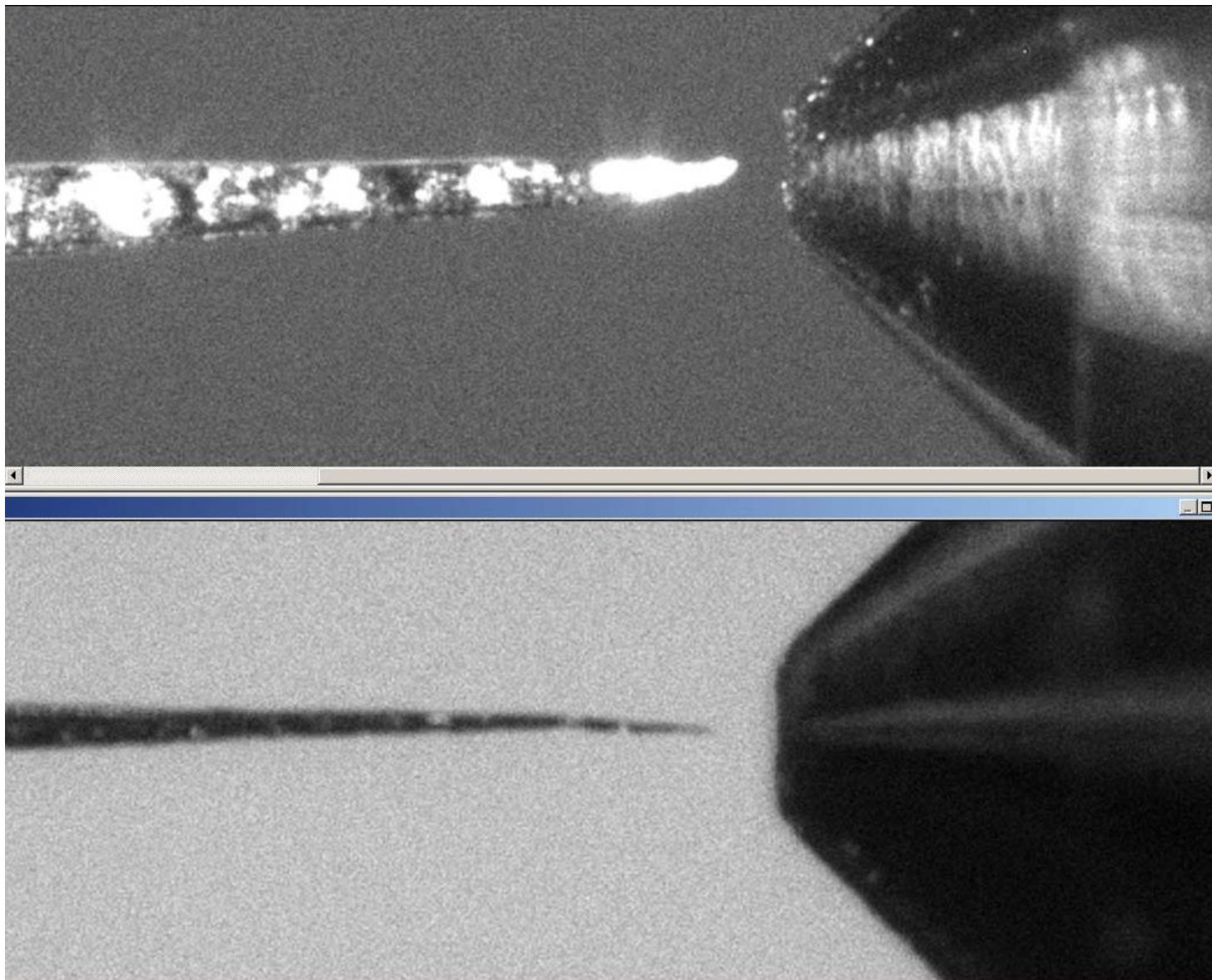
Nb Superconducting RF cavity

- Performance of Nb cavity
 - Oxygen
 - Hydrogen
- Oxide on surface
 - Chemistry, thickness
- Oxygen in bulk
 - Concentration, location, and form

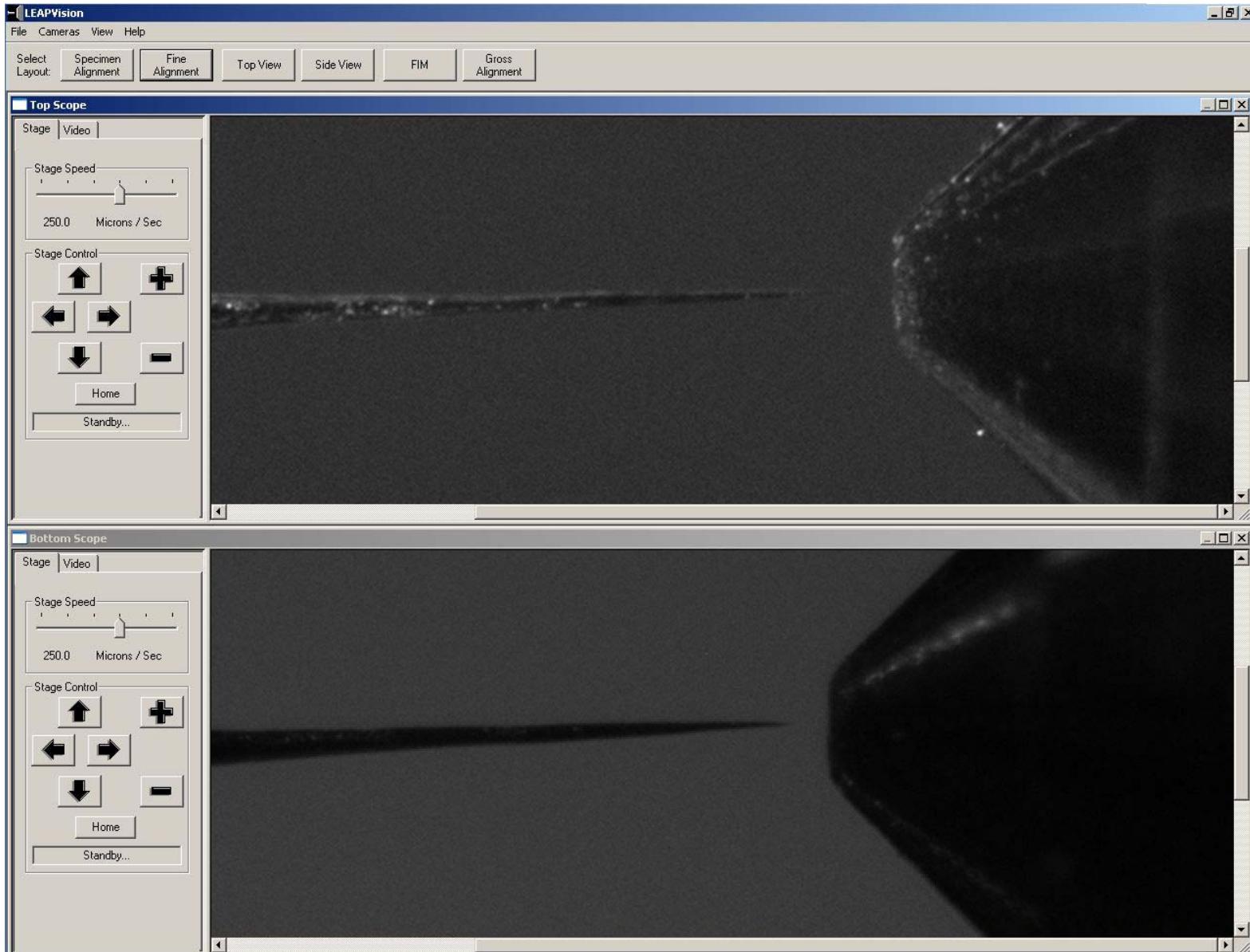
Sample preparation

- $0.5 \times 0.6 \times 10 \text{ mm}^3$ Nb blanks
- $0.2 \times 0.3 \times 10 \text{ mm}^3$ Nb blanks
- $0.2 \times 0.2 \times 10 \text{ mm}^3$ Nb blanks
- Electropolishing
- HF : $\text{H}_2\text{SO}_4 = 1 : 10$
 - With a few drops of HNO_3
- Use DC voltage
 - Start at 31 V ~ decrease to 10 V
 - Minimize pulsing
- Rinse with deionized water

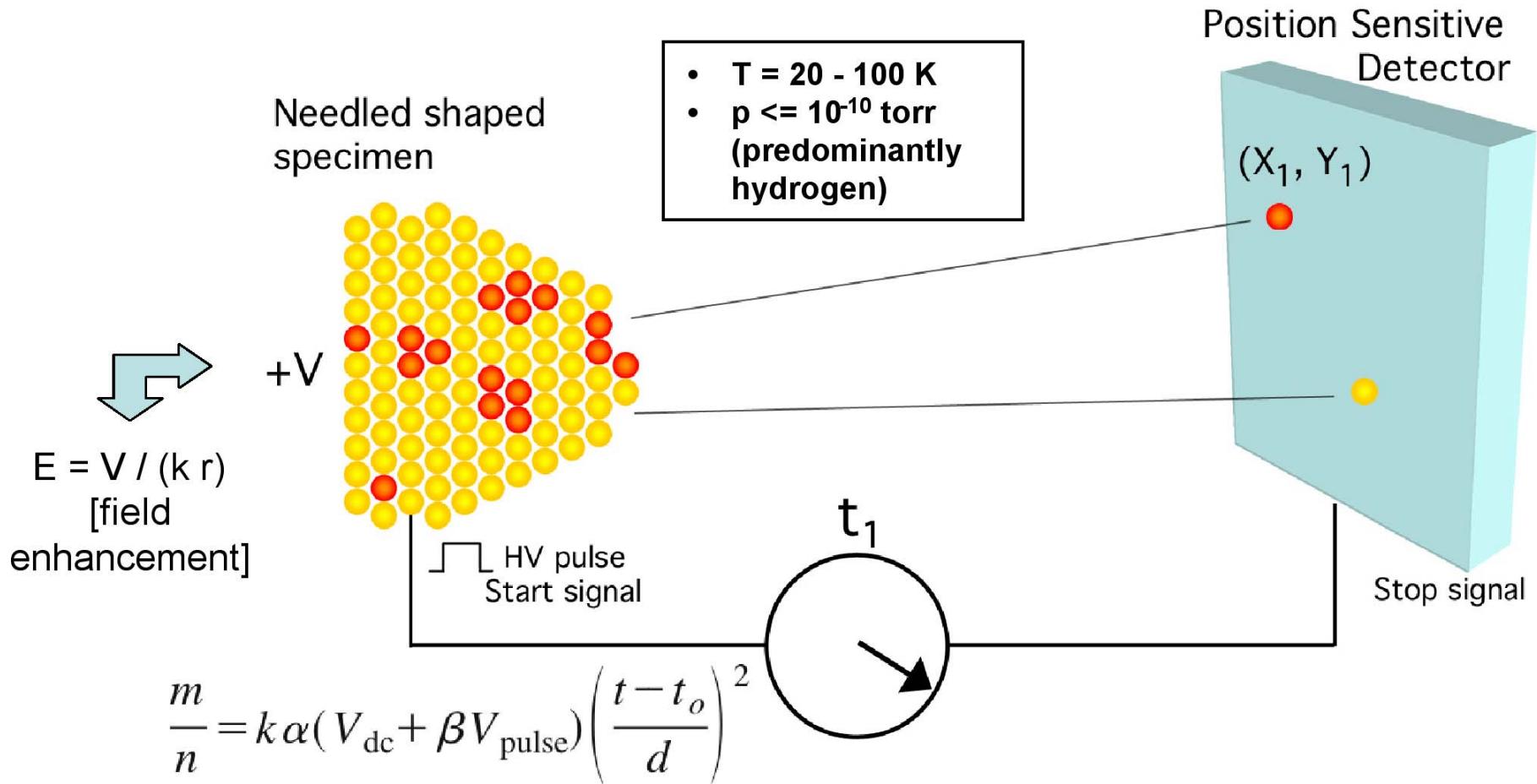
Nb tip after EP 1



Nb tip after EP 2



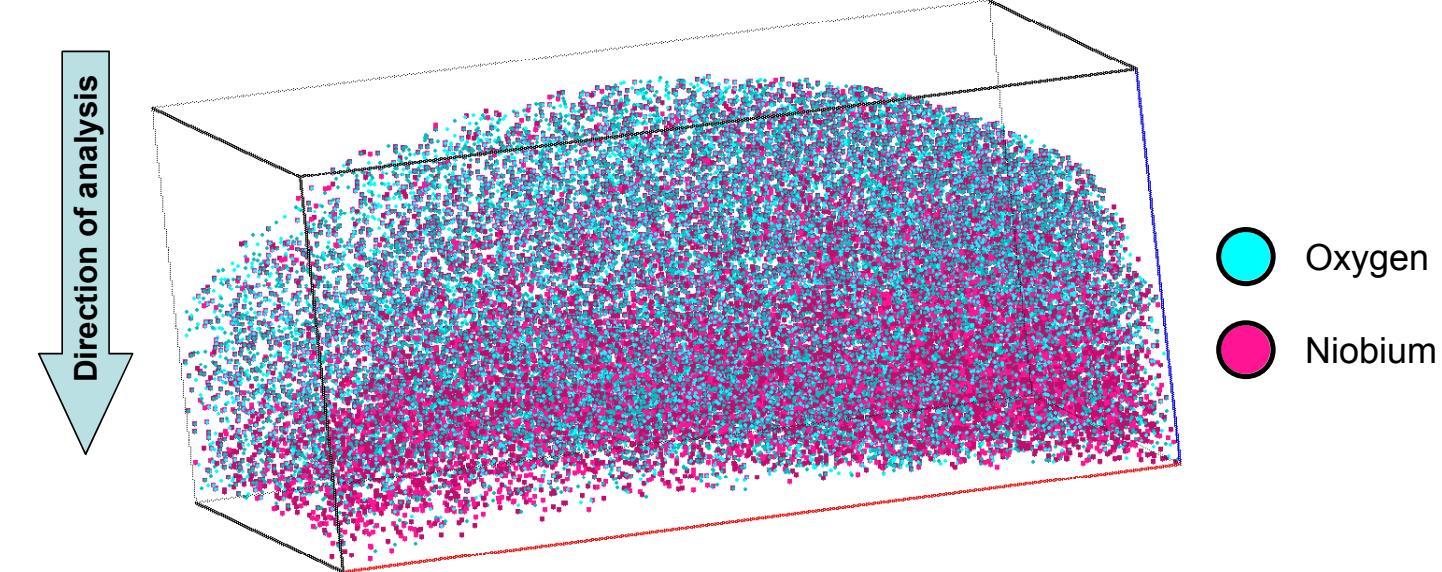
Atom-probe tomography (APT)



- Coordination of ions (x, y, and z):
Allows for the three-dimensional reconstruction of real space
- times-of-flight: Mass-to-charge ratio= identifying the elements

$$neV_0 = \frac{1}{2} m \frac{d^2}{t^2} \quad \frac{m}{n} = 2eV_0 \frac{t^2}{d^2}$$

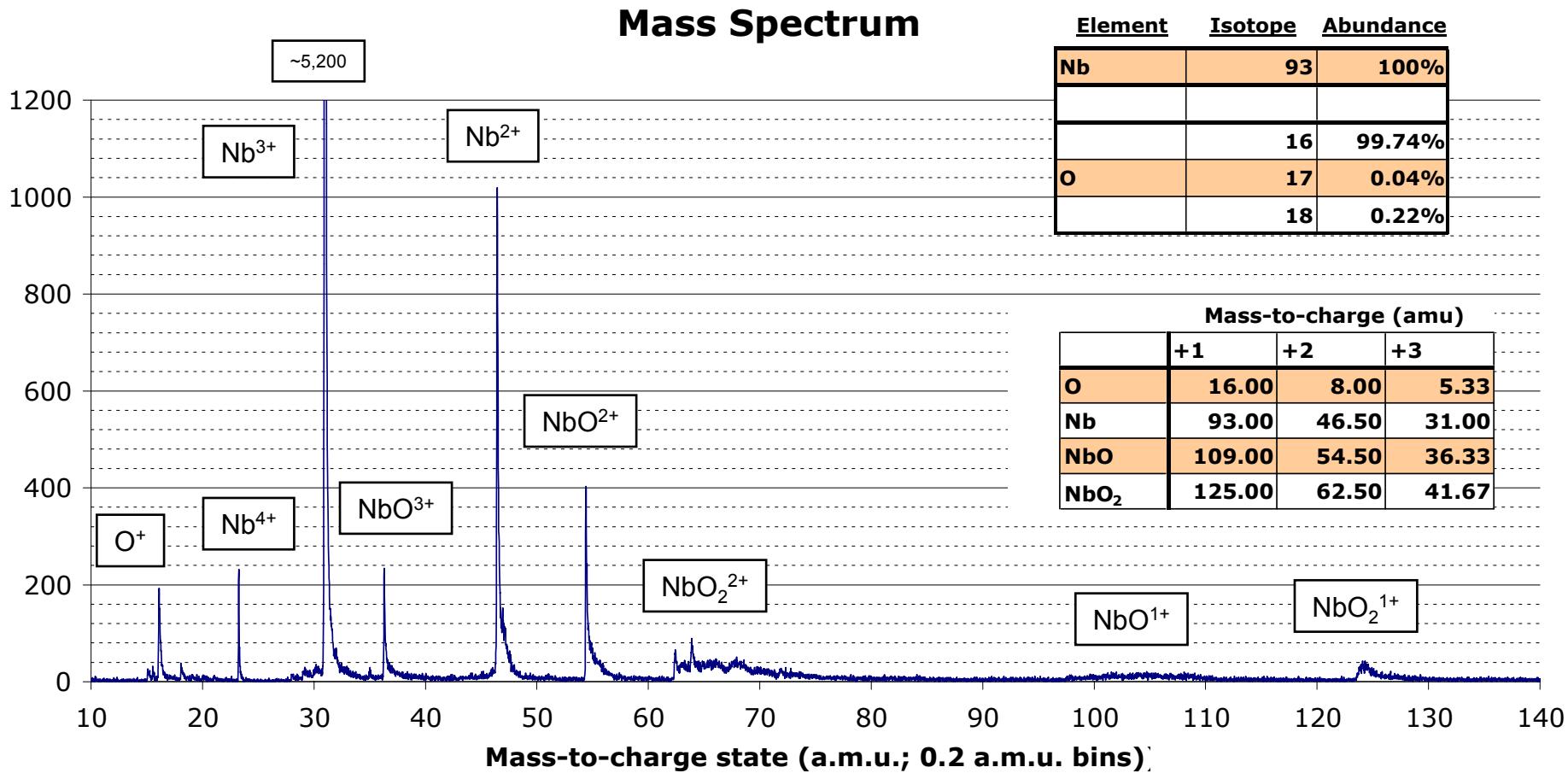
- Atomic resolution



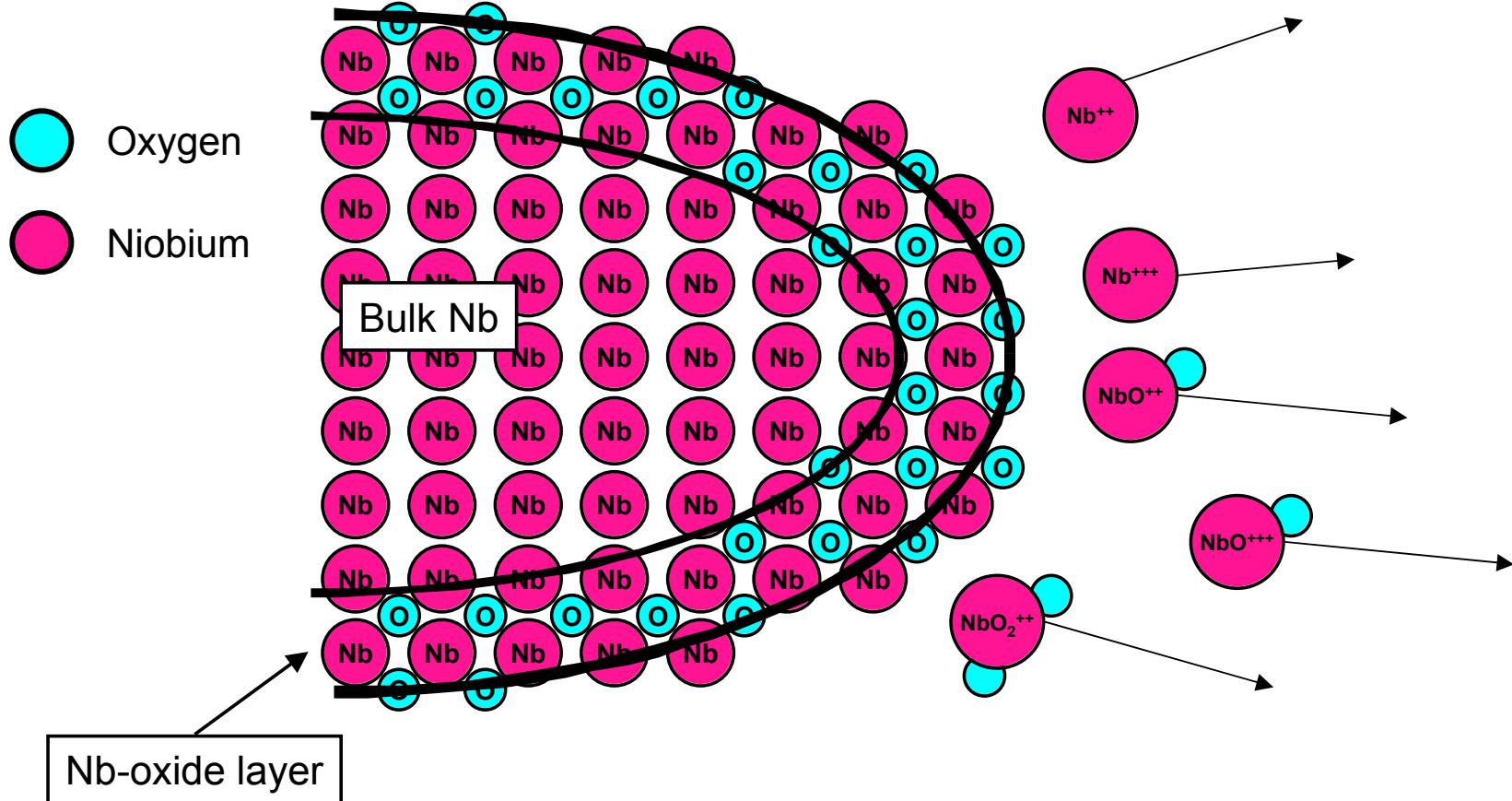
Previous Results

- $23 \times 21 \times 11 \text{ nm}^3$
- 112 K atoms
- Nb magenta
- O cyan

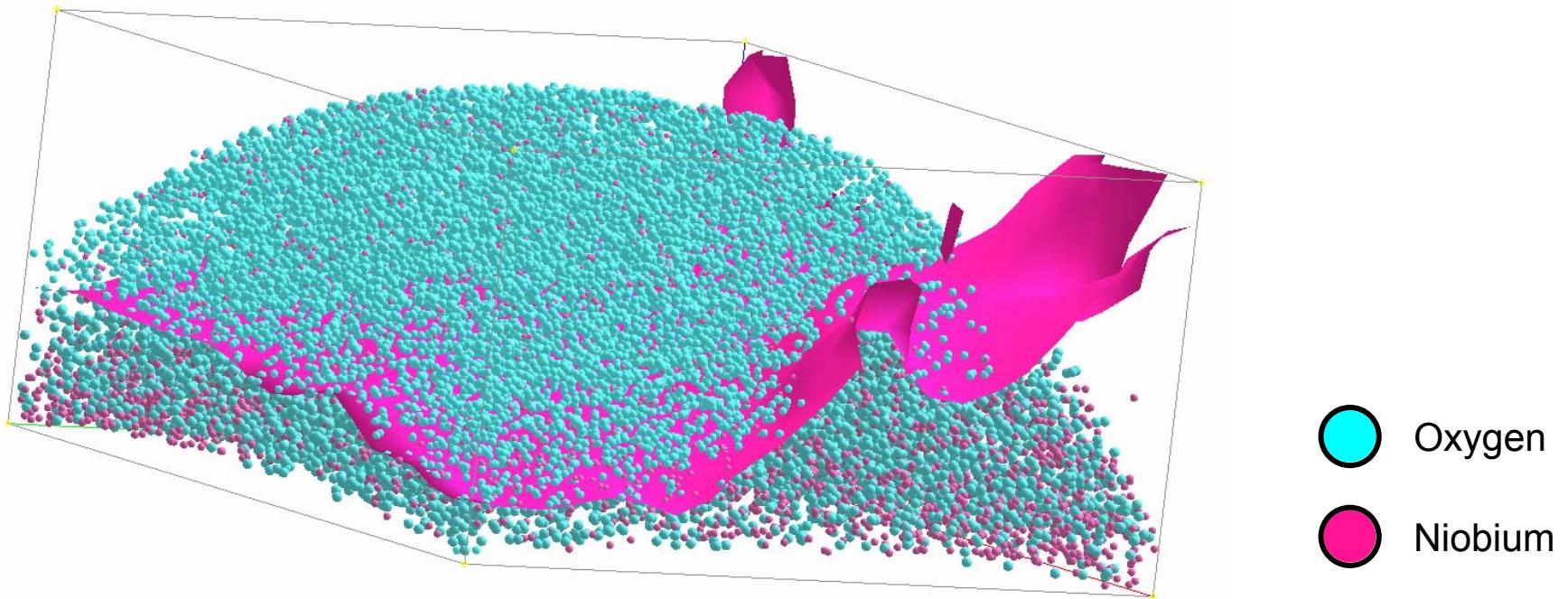
Mass spectrum



Evaporation of complex ions

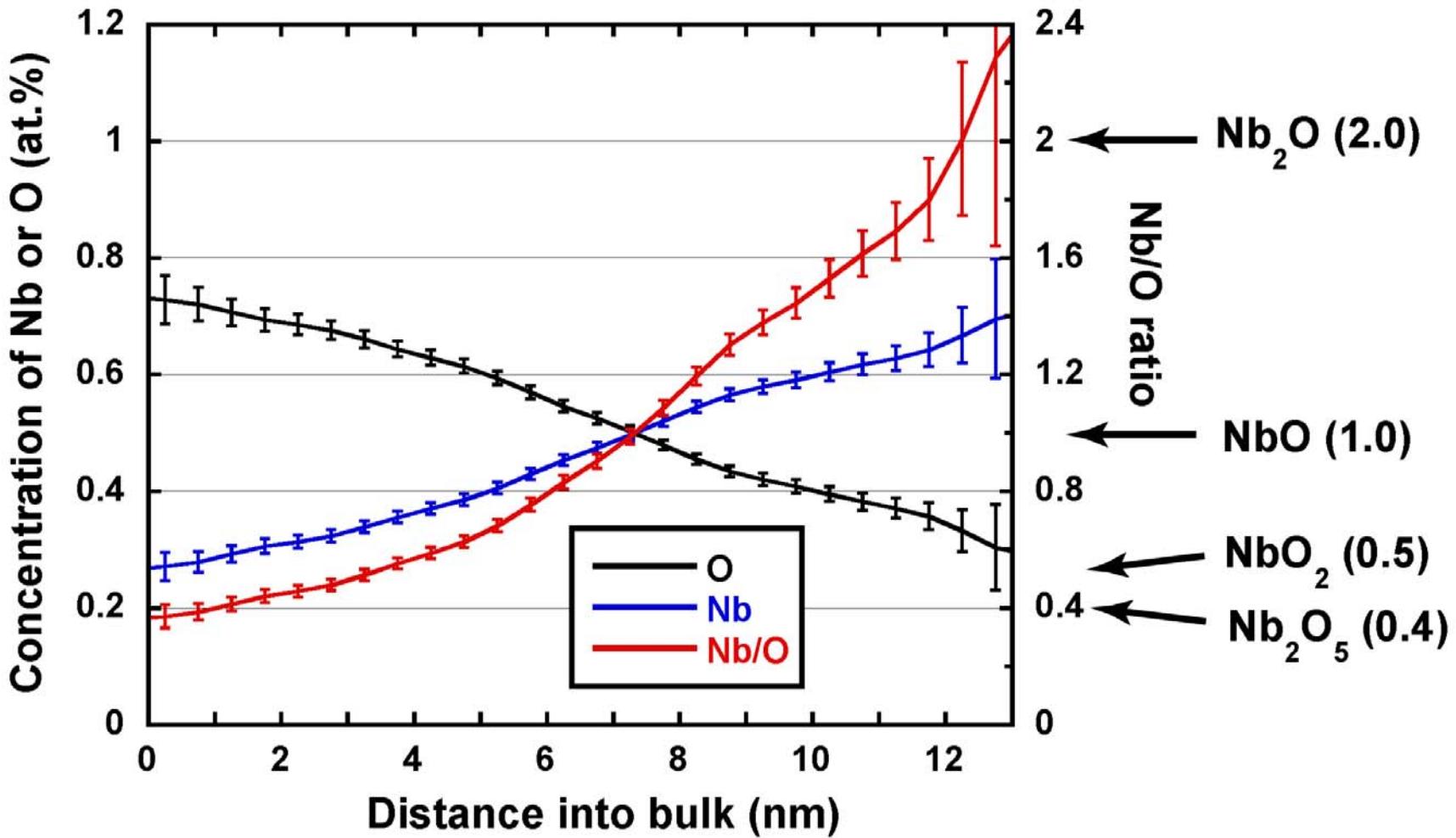


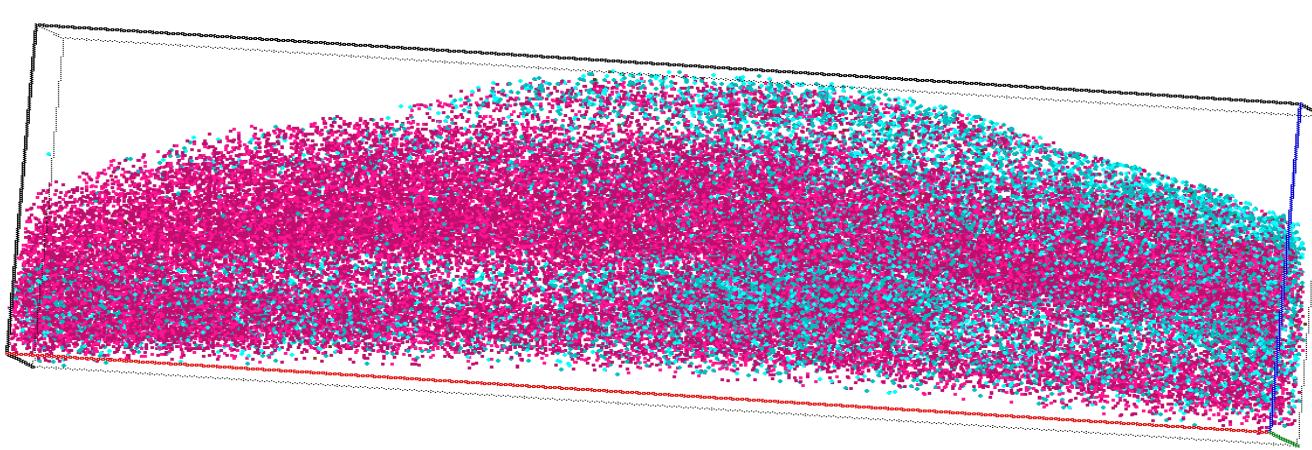
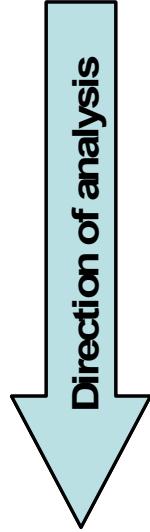
Isoconcentration surface



- Identify the interface
- 30 at.% Nb isocentration surface

Proximity Histogram

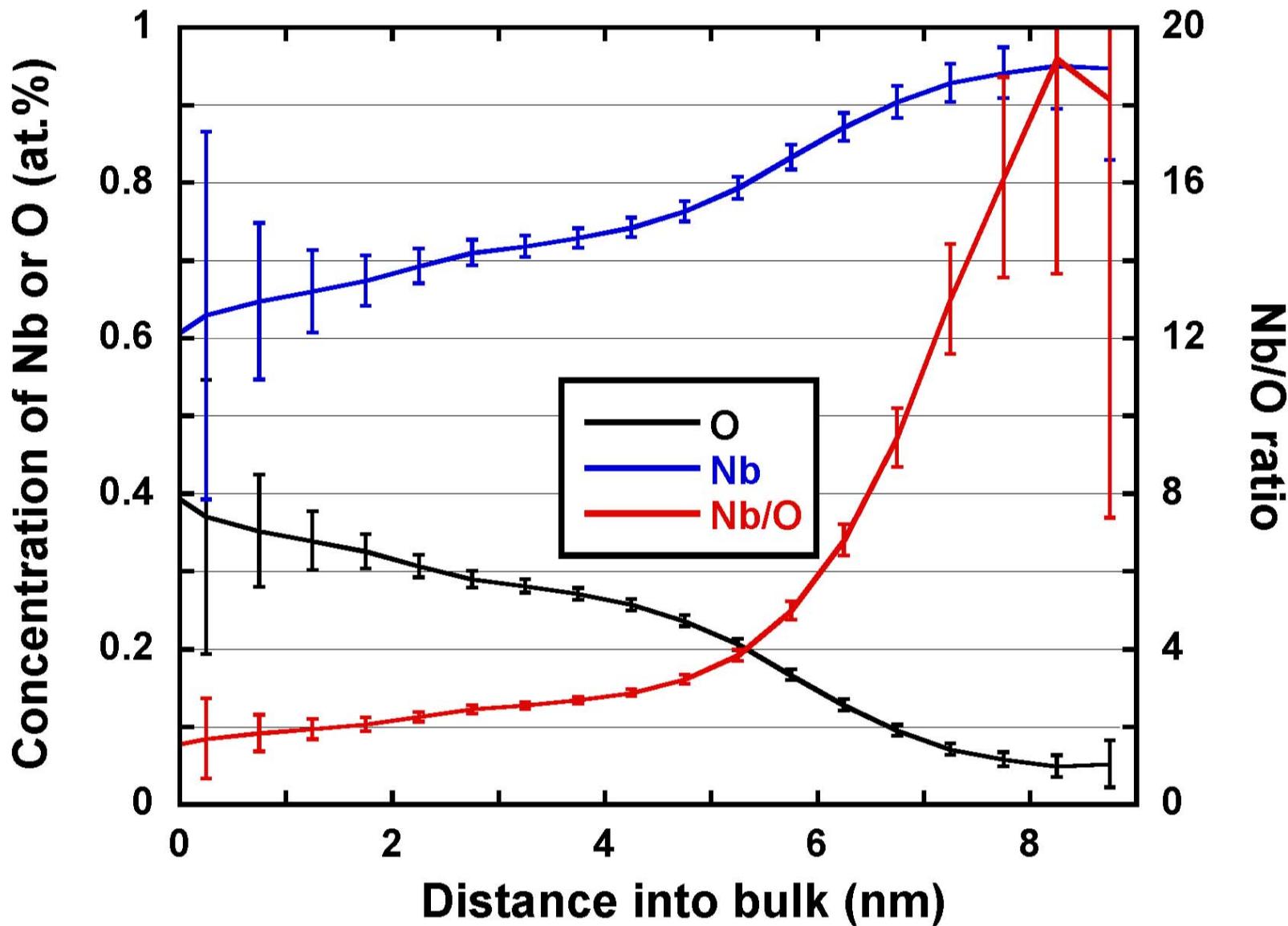




Previous Results

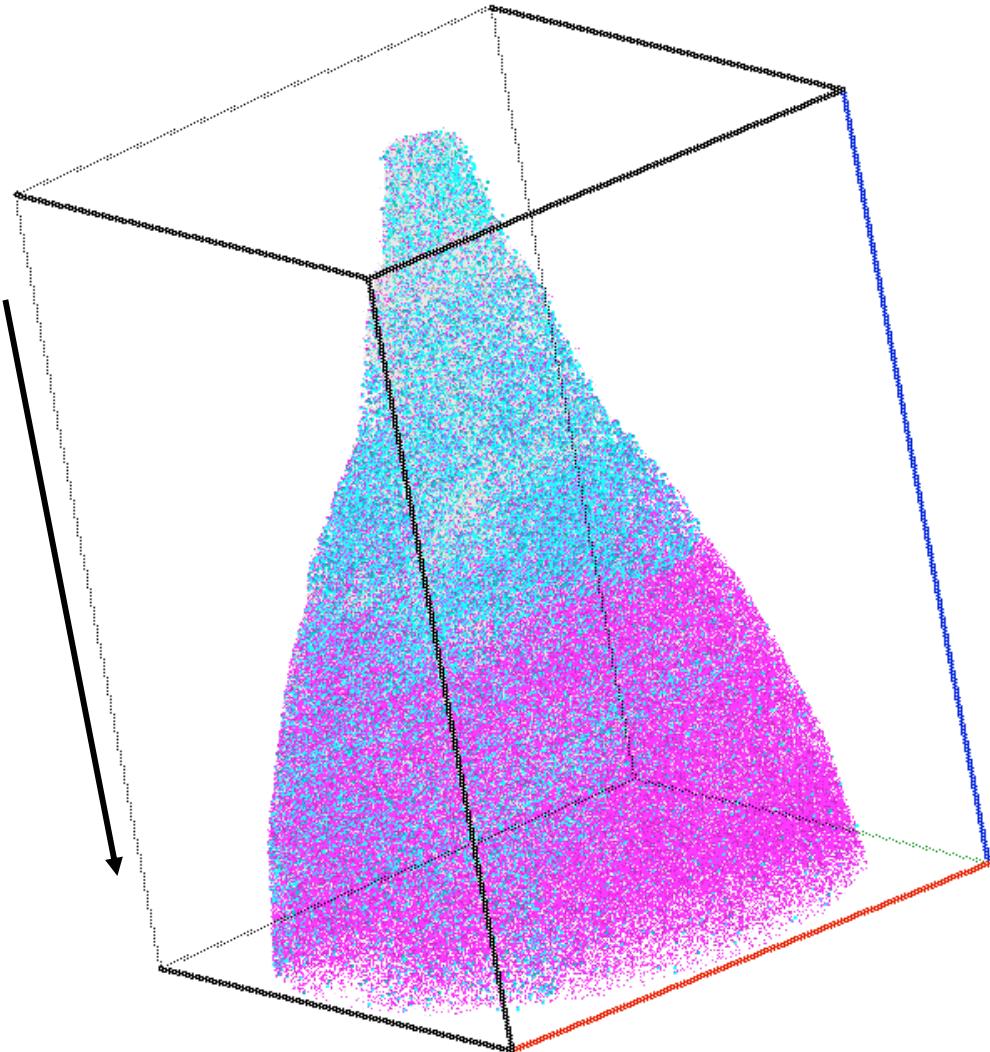
- $25 \times 25 \times 6 \text{ nm}^3$
- 75 K atoms
- Nb magenta
- O cyan

Proximity Histogram



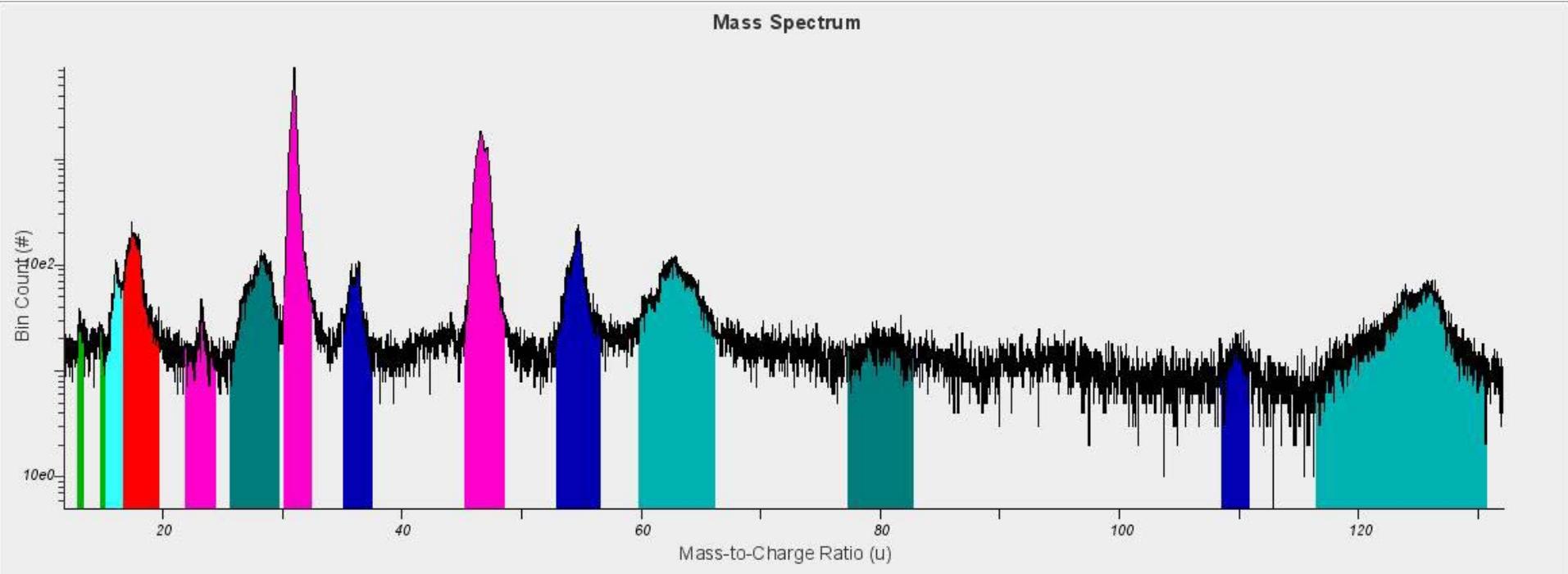
1st Results

- $25 \times 26 \times 48 \text{ nm}^3$
- 0.5 M atoms
- Nb magenta
- O cyan



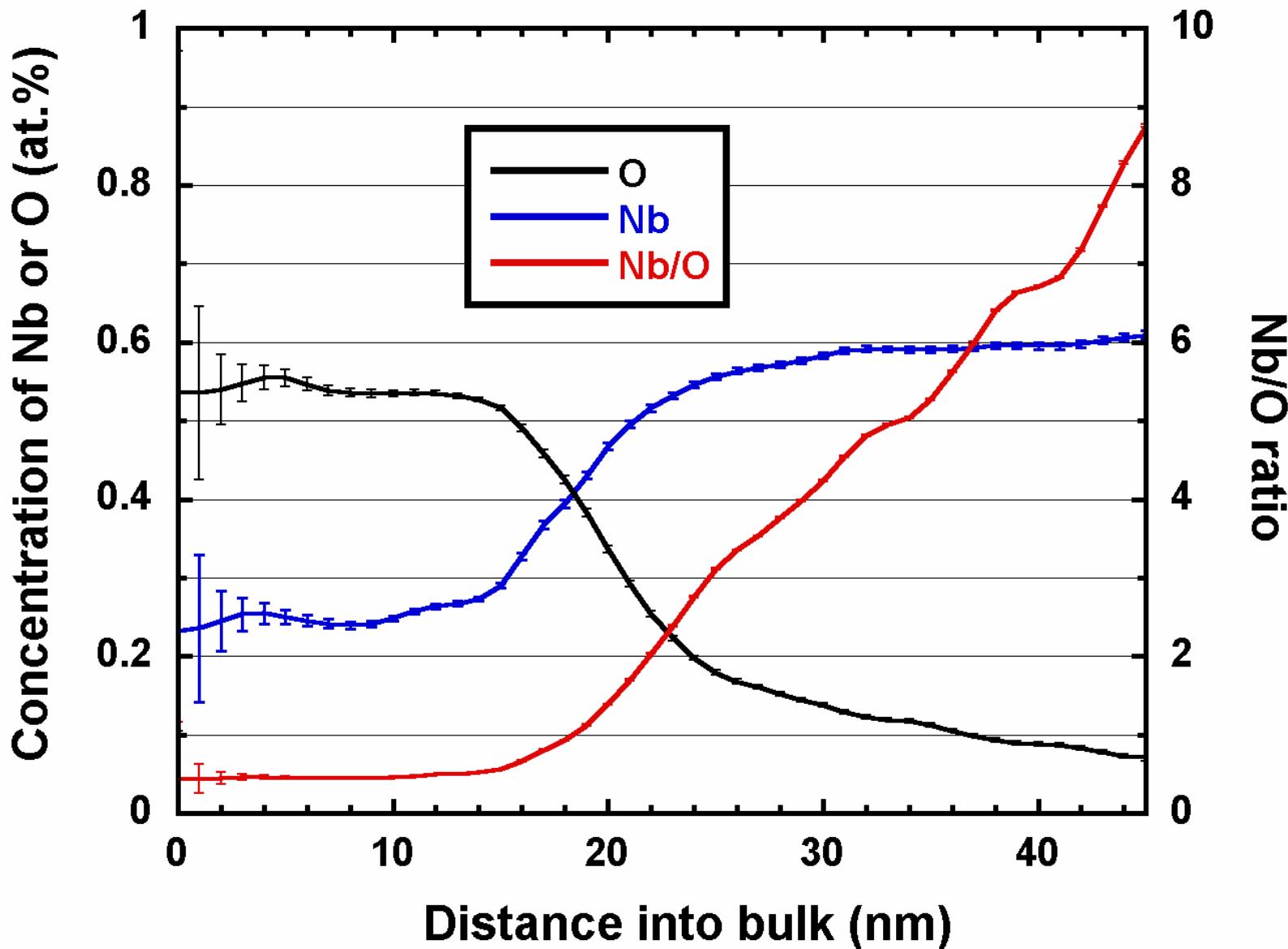
Analysis
direction

Mass spectrum



- NH^+ : 15 O^+ : 16 H_2O^+ : 18
- Nb^{4+} : 23.25 $\text{NbOH}_6^{4+}/\text{NbO}_3\text{H}_3^{5+}$: 29
- Nb^{3+} : 31 NbO^{3+} : 36.3
- Nb^{2+} : 46.5 NbO^{2+} : 54.5 NbO_2^{2+} : 62.5
- NbO^+ : 109 NbO_2^+ : 125

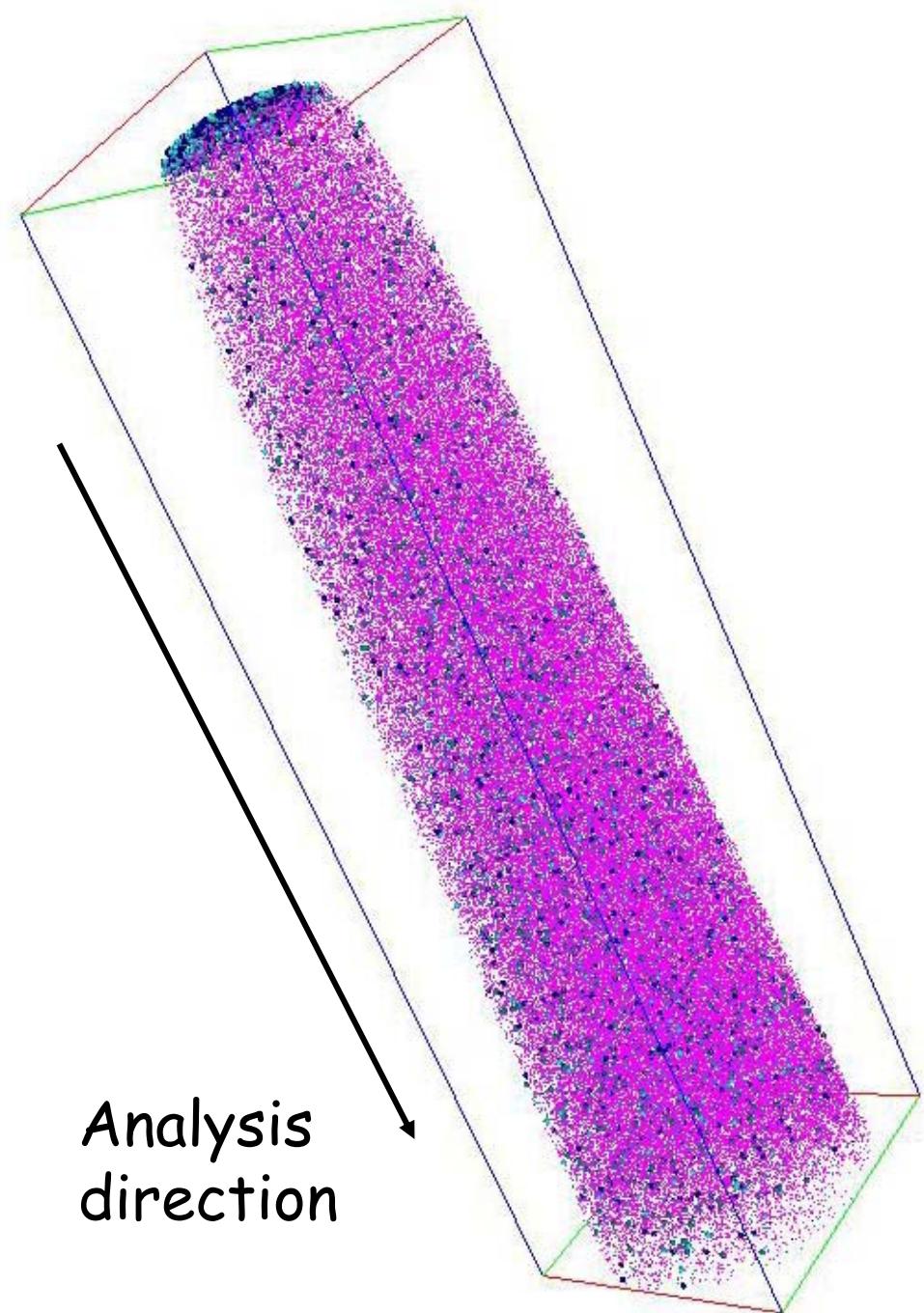
Proximity Histogram



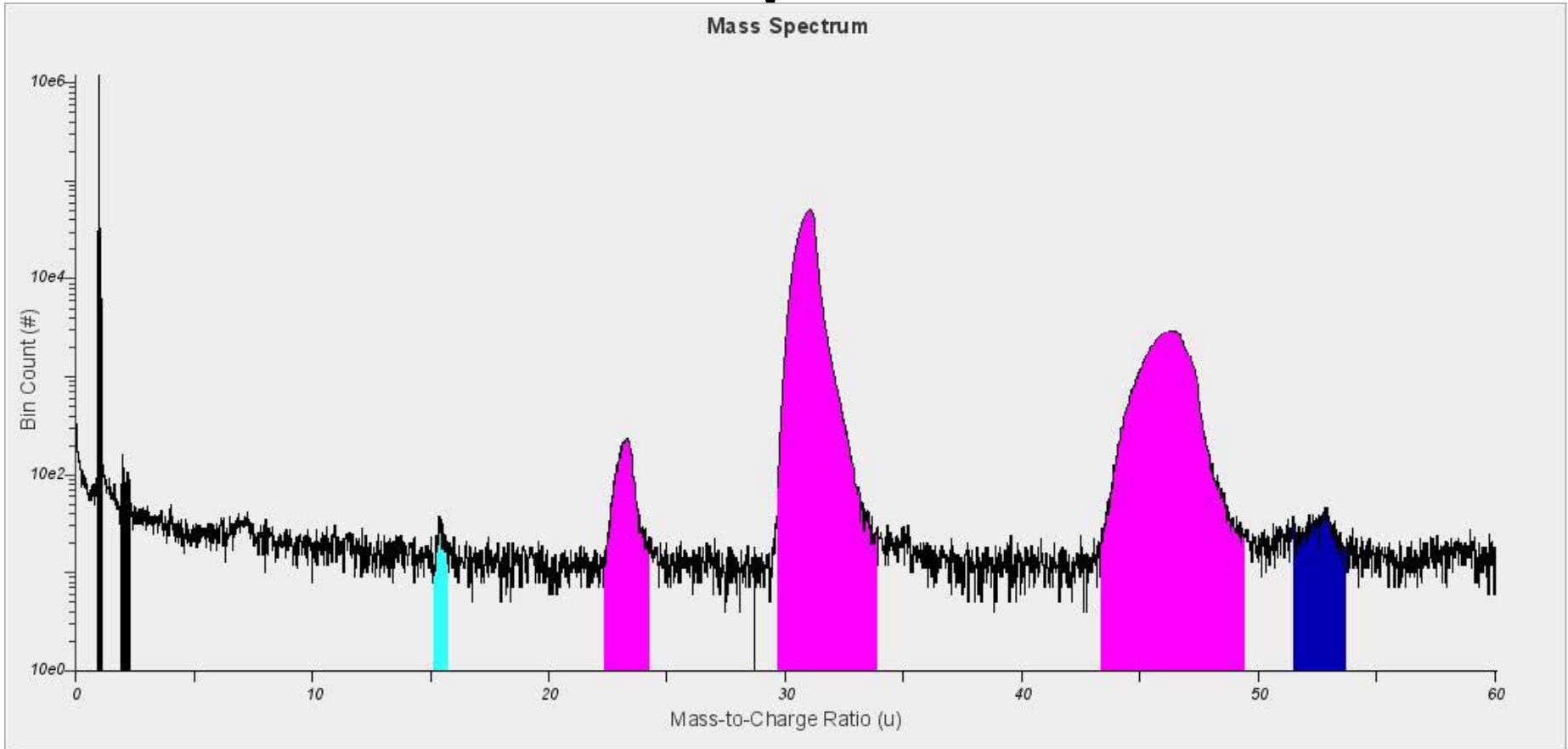
2nd Results

- $38 \times 40 \times 145 \text{ nm}^3$
- 3.8 M atoms
- Nb magenta
- O cyan

Analysis
direction

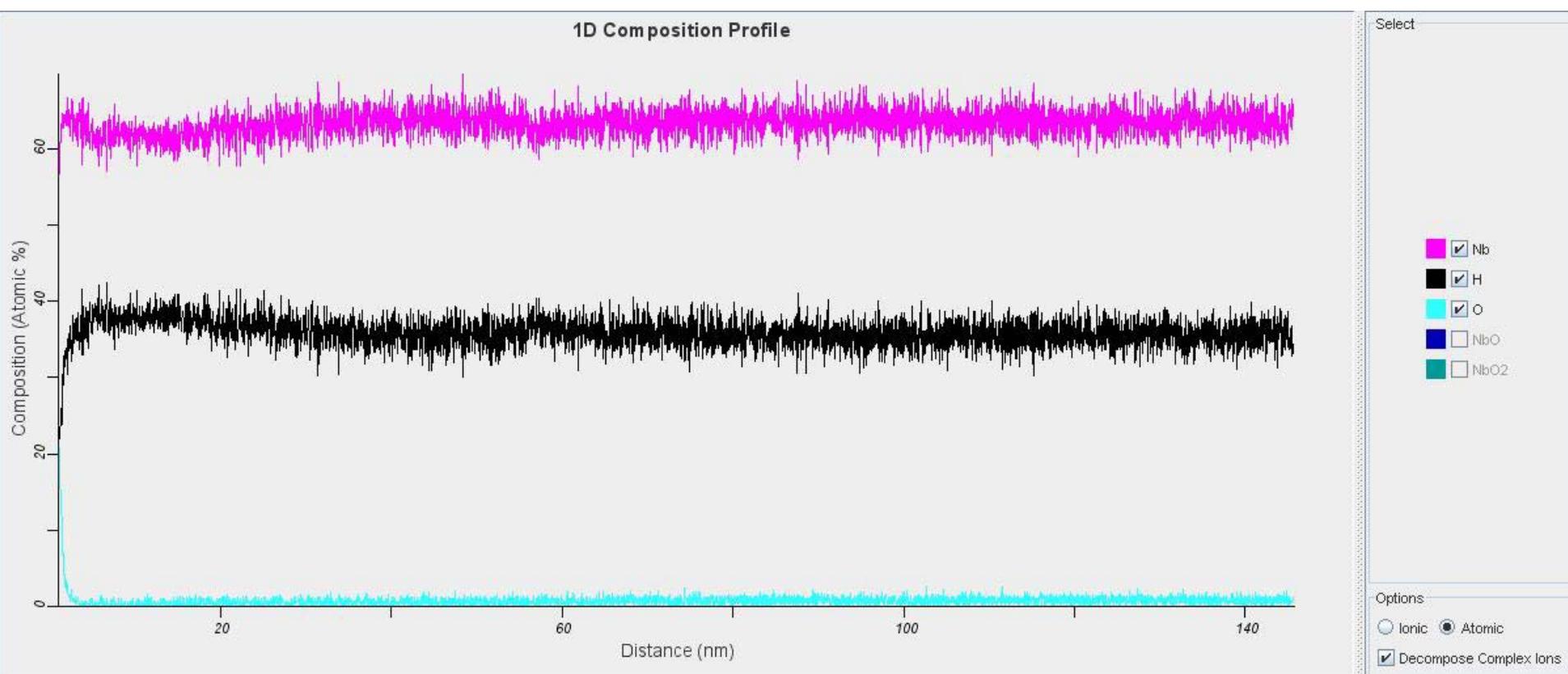


Mass spectrum

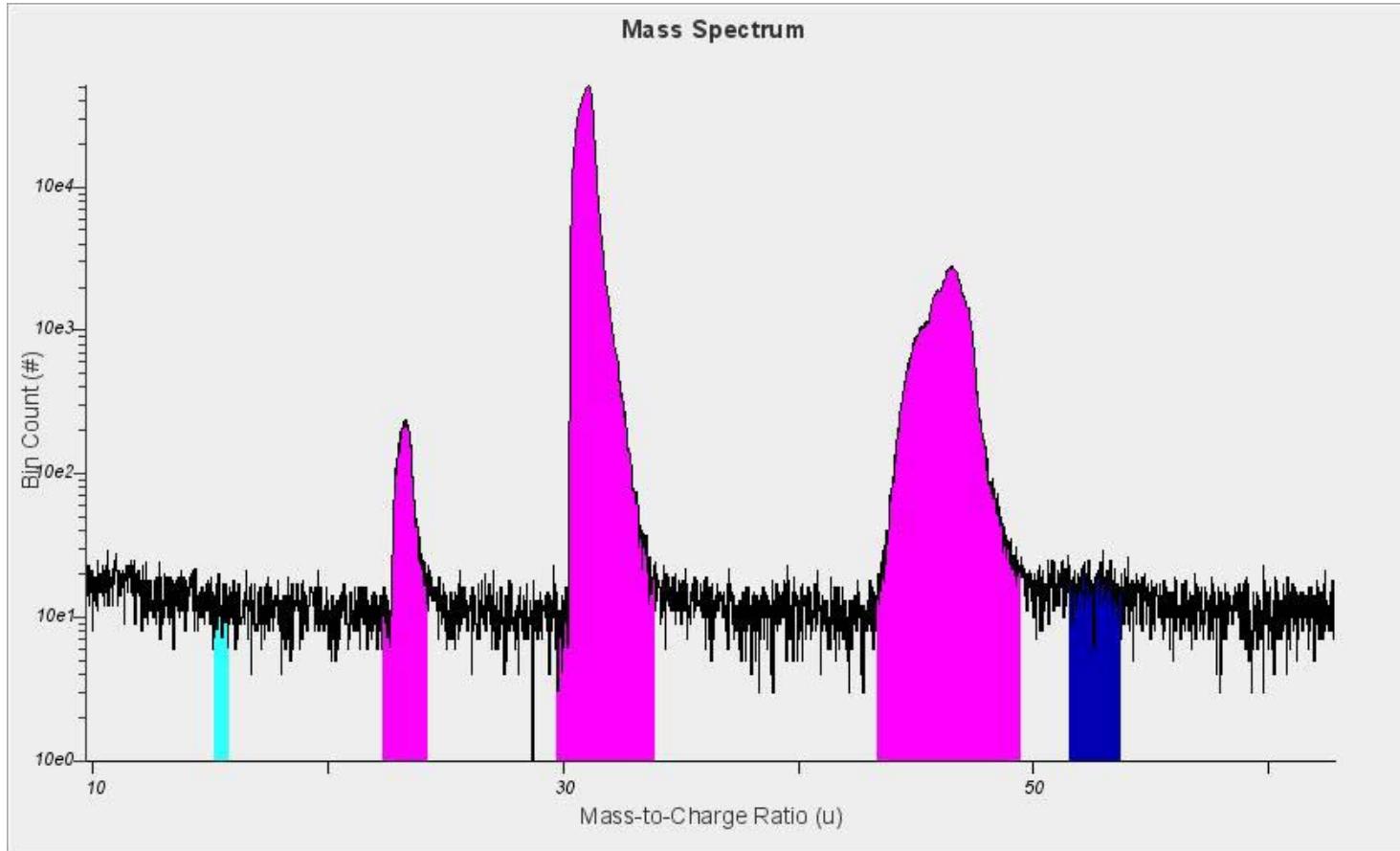


- O^+ : 16 Nb^{4+} : 23.25 Nb^{3+} : 31
- Nb^{2+} : 46.5 NbO^{2+} : 54.5

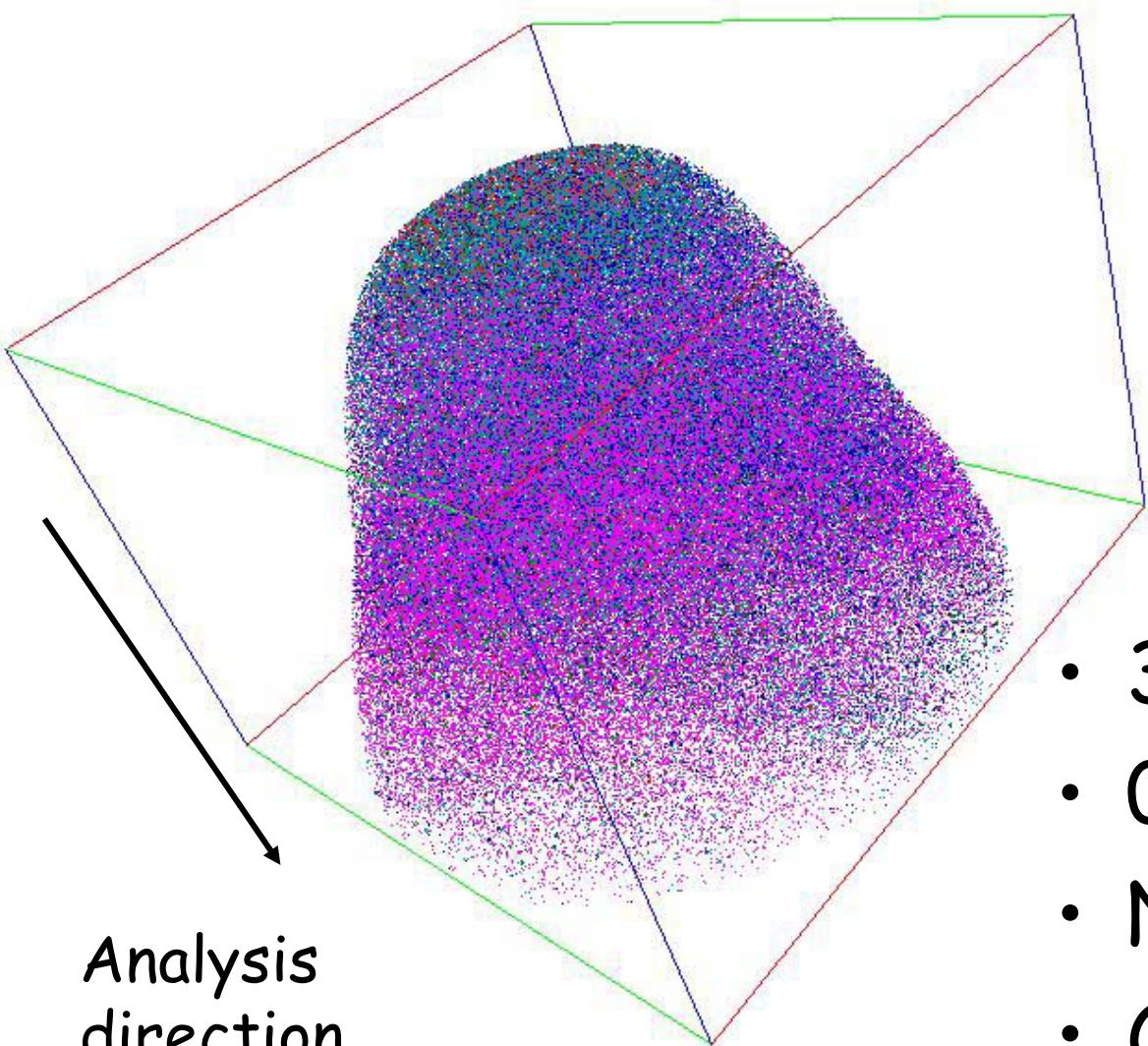
Composition profile



Mass spectrum of bulk



- No oxygen peak
- Less than 50 ppm

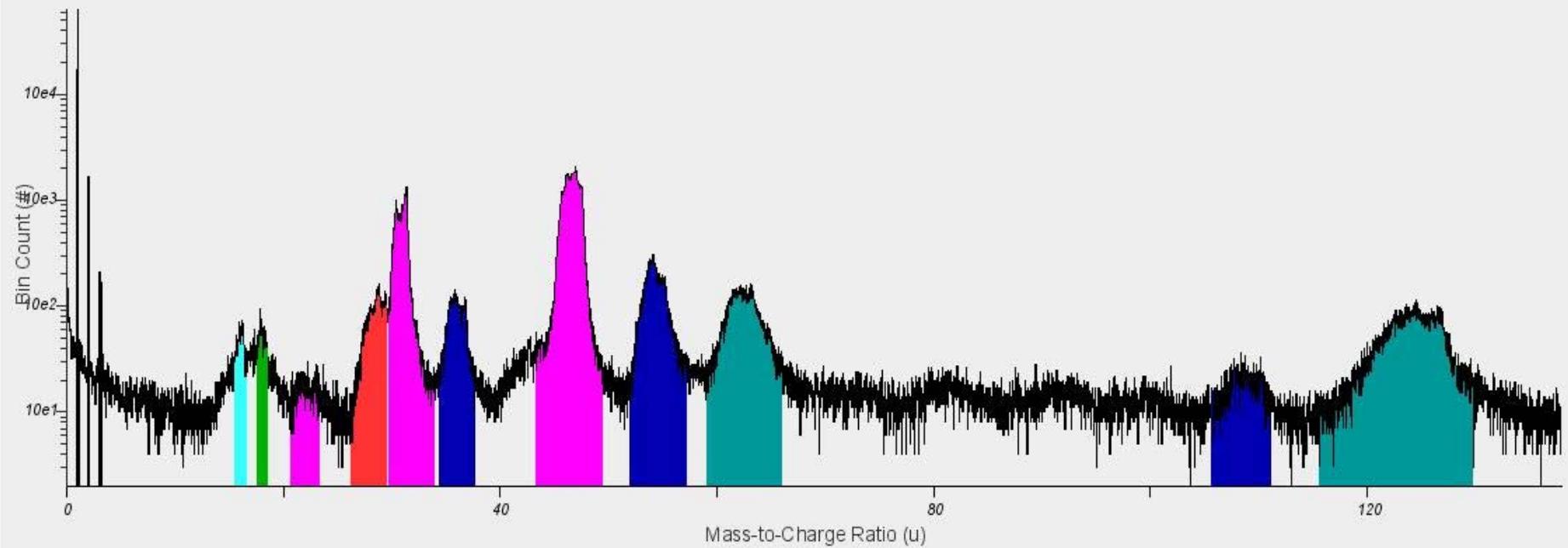


3rd Results

- $36 \times 35 \times 26 \text{ nm}^3$
- 0.5 M atoms
- Nb magenta
- O cyan

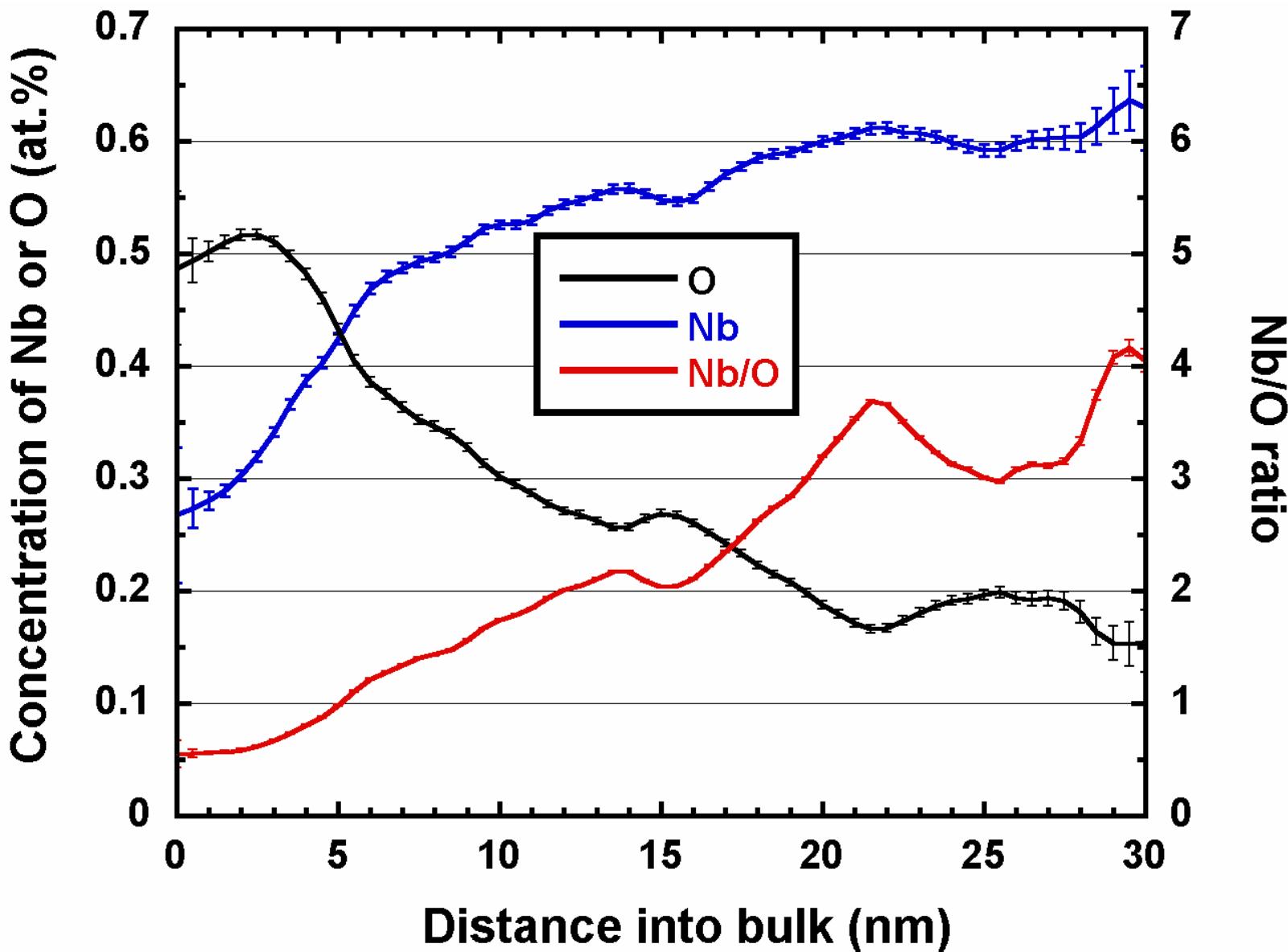
Mass spectrum

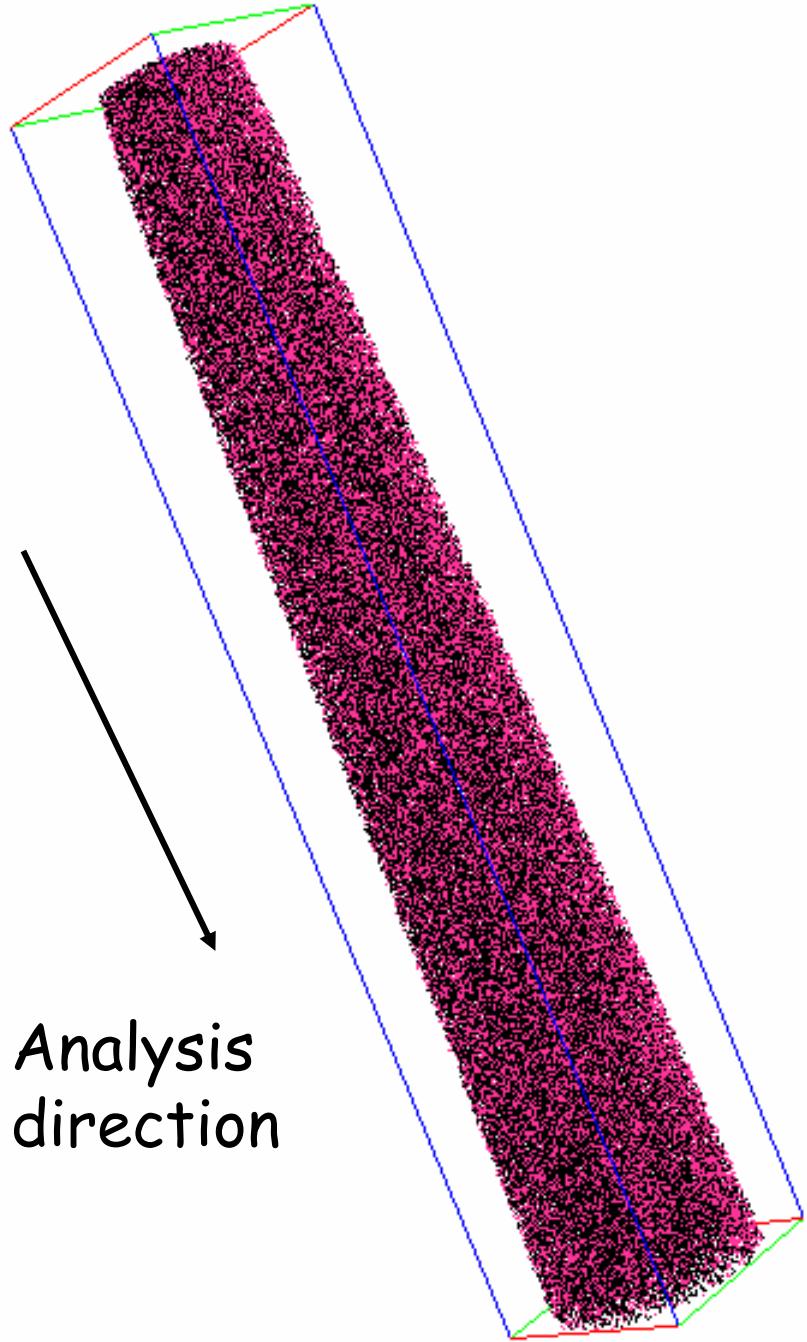
Mass Spectrum



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- Nb^{4+} : 23.25 $NbOH_6^{4+}$ / $NbO_3H_3^{5+}$: 29
- Nb^{3+} : 31 NbO^{3+} : 36.3
- Nb^{2+} : 46.5 NbO^{2+} : 54.5 NbO_2^{2+} : 62.5
- NbO^+ : 109 NbO_2^+ : 125

Proximity Histogram



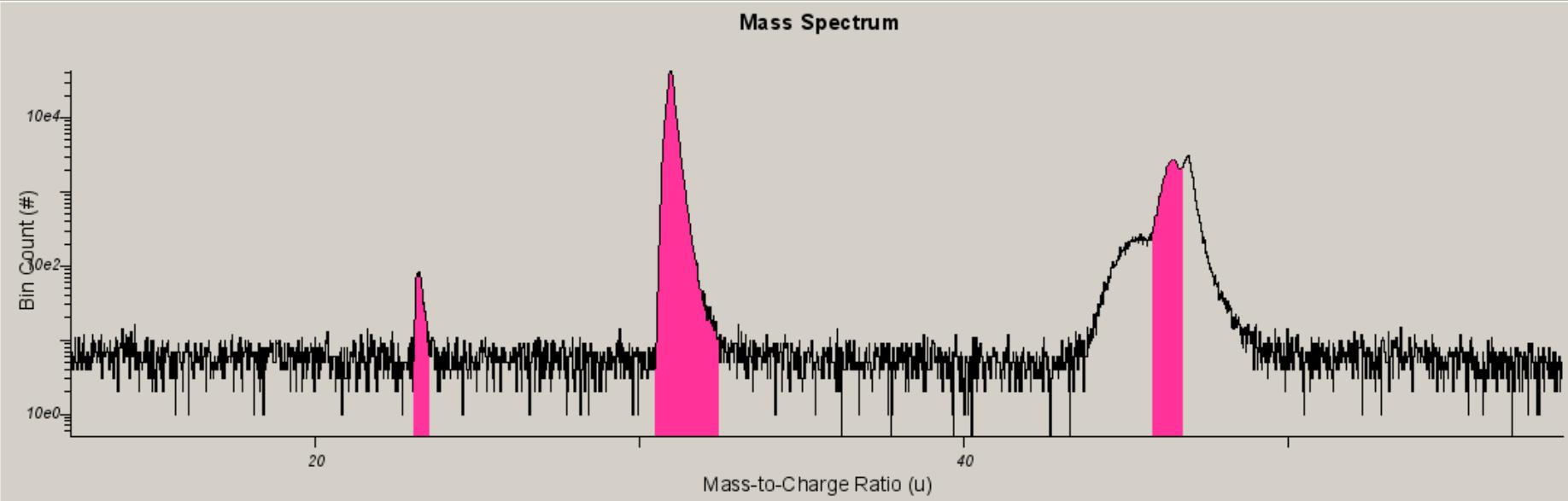


Analysis
direction

4th Results

- $21 \times 21 \times 123 \text{ nm}^3$
- 0.9 M atoms
- Nb magenta
- H black
- No O detected

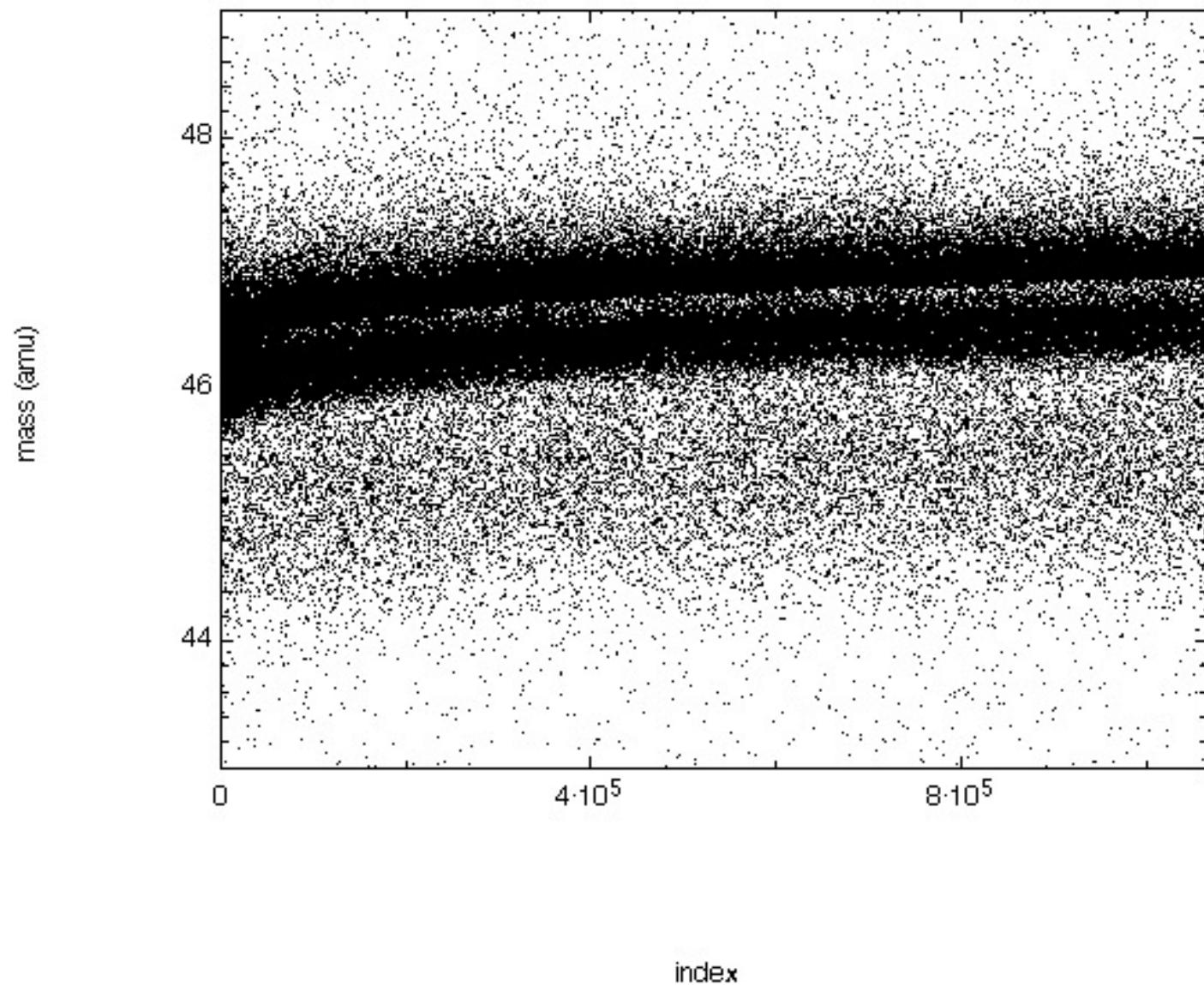
Mass spectrum

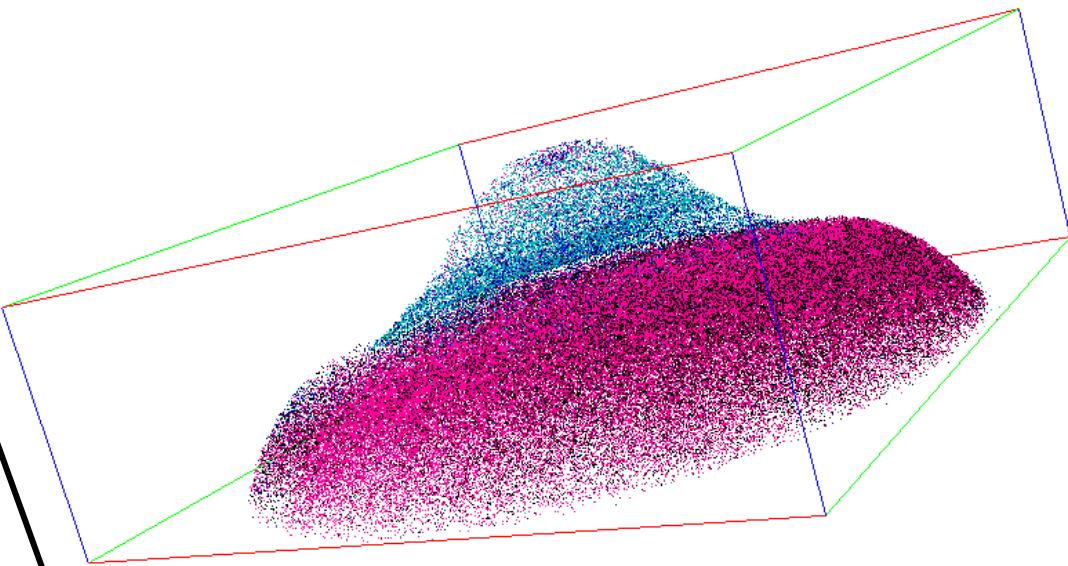


- Nb^{4+} :23.25
- Nb^{3+} :31
- Nb^{2+} :46.5

Mass chart

output.atomlist

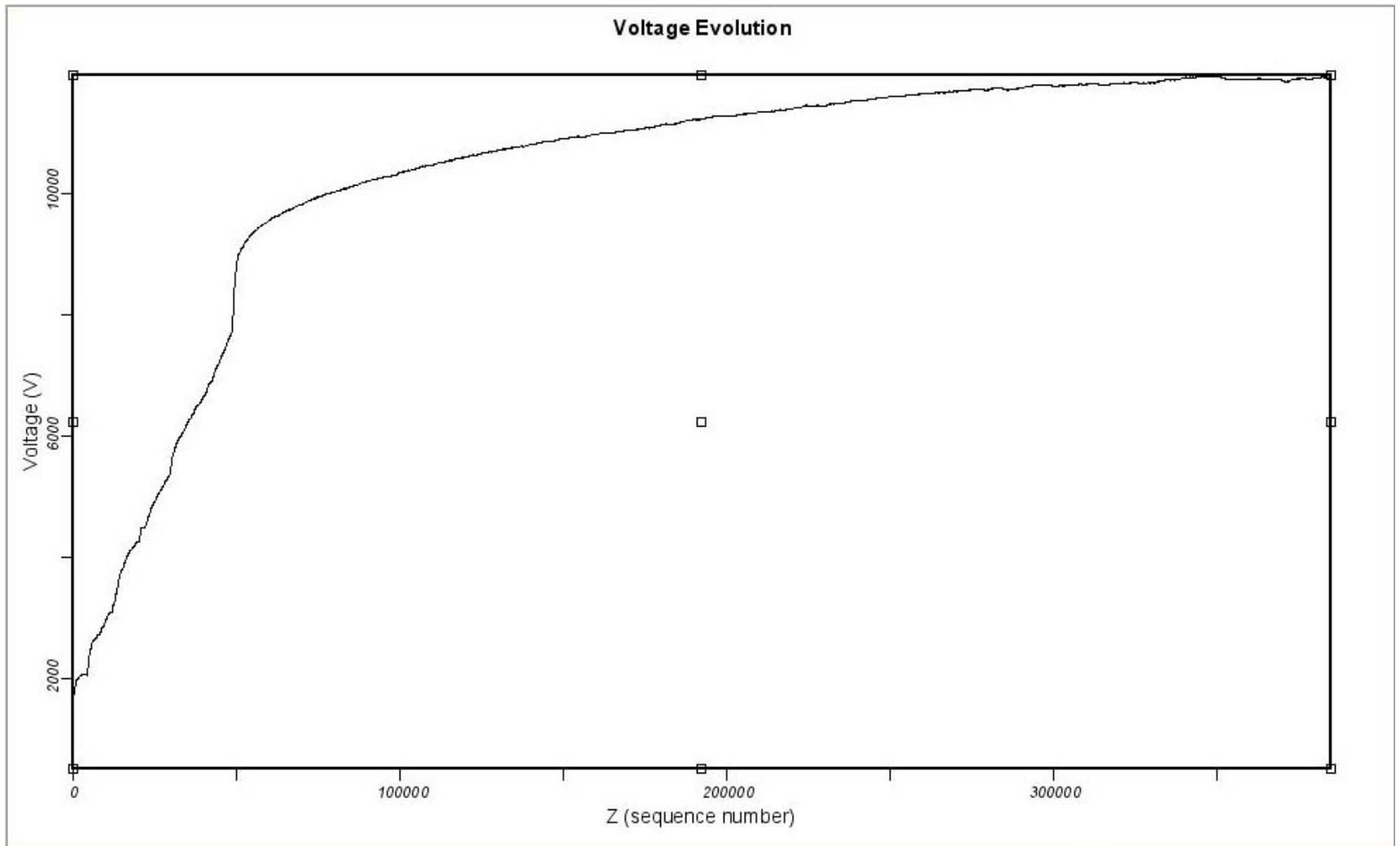




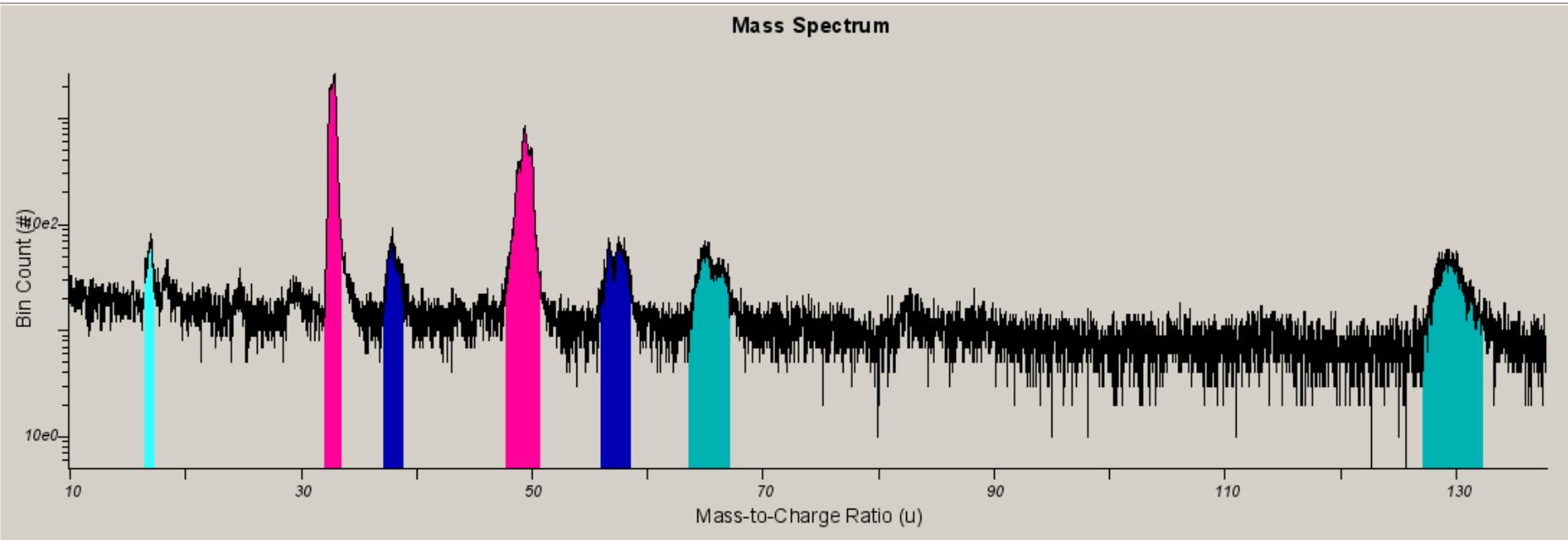
5th Results

- $21 \times 21 \times 123 \text{ nm}^3$
- 0.9 M atoms
- Nb magenta
- H black
- No O detected

Voltage curve



Mass spectrum



- O^+ : 16
- Nb^{4+} : 23.25
- Nb^{3+} : 31 NbO^{3+} : 36.3
- Nb^{2+} : 46.5 NbO^{2+} : 54.5 NbO_2^{2+} : 62.5
- NbO_2^+ : 125

Summary

tip	oxide	interface	transition
Previous results	2 nm -Nb ₂ O ₅	10 nm	smooth
	2 nm-Nb ₂ O	8 nm	rapid
1st	15 nm-Nb ₂ O ₅	15 nm	smooth
2nd	0 nm	0 nm	Bulk Nb
3rd	5 nm-Nb ₂ O ₅	15 nm	smooth
4th	0 nm	0 nm	Bulk Nb
5th	N/A	N/A	

Conclusions

- Atomic scale investigation of surface oxide to bulk Nb
 - First time to reach bulk Nb
 - Surface: possibly Nb_2O_5
 - Thicker than expected: $> 5 \text{ nm}$
 - High level of O below surface oxide: $\sim 20\%$
 - High level of H in oxide and bulk: $\sim 20\%$
 - Scattered results
 - Electropolishing dependent
 - Need better statistics

Next step

- TEM investigation
 - Structure of oxide
- Use FIB for sample preparation
- Investigation of heat-treated Nb samples